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<p>I. Polysilane High Polymers and Oligomers. a) Light-scattering studies of molecular weight and chain conformations were carried out for six polysilane samples. b) Silicon-29 NMR spectroscopy was employed to study configurations of polysilane polymers and oligomers. c) A new class of substances, poly(disilanylene-co-ethynylene) copolymers, were synthesized which show properties indicating σ-π electron conjugation.</p> <p>II. Divalent and Multiply-Bonded Silicon Compounds. a) Complexes of silylenes, $R_2Si:$, with various Lewis bases have been observed spectroscopically. b) Reactions of disilenes ($R_2Si=SiR_2$) with nitro compounds, nitroso compounds, azides and oxygen have been studied, leading to previously unknown types of cyclic silicon compounds. c) A new, very stable disilene, 1,2-dimesityl-1,2-bis(1-adamantyl)disilene, was synthesized and investigated. d) The first silandiimines, $RN=Si=NR$, have been made and observed at low temperatures. e) Stable silan-amidines, $RNH=Si(X)=NR$, and silanamide anions, $RN^-=Si(X)=NR^-$, have been synthesized and studied.</p>			
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UNCLASSIFIED

Organic silicon compounds

Final Technical Report

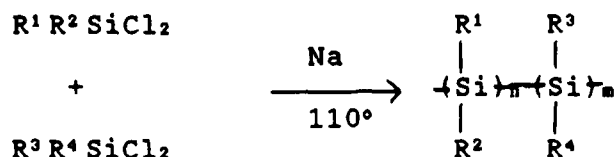
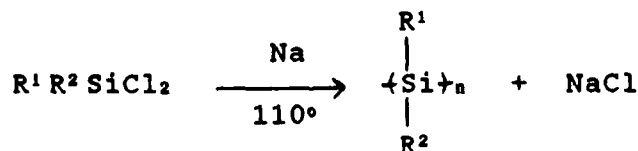
CHEMICAL REACTIONS AND PROPERTIES OF ORGANOSILICON COMPOUNDS RELATED TO NEW MATERIALS

Period Covered: October 1, 1985 - September 30, 1988

In this report will be described the most significant findings from research under AFOSR sponsorship during the three-year period of this Contract. The areas to be covered are: I. Polysilane High Polymers and Oligomers; and II. Divalent and Multiply Bonded Silicon Compounds, including A. Silylenes, $RR'Si$; B. Disilenes, $RR'Si=SiRR'$; and C. Silanediimines, $RN=Si=NR$ and Silanamidines, $RNH-Si(X)=NR$.

I. Polysilane High Polymers and Oligomers

The polysilane polymers are linear polymers in which the "backbone" polymer chain is made up entirely of silicon atoms. They are generally synthesized by sodium condensation of diorganodichlorosilanes. Either homopolymers or copolymers can be made:



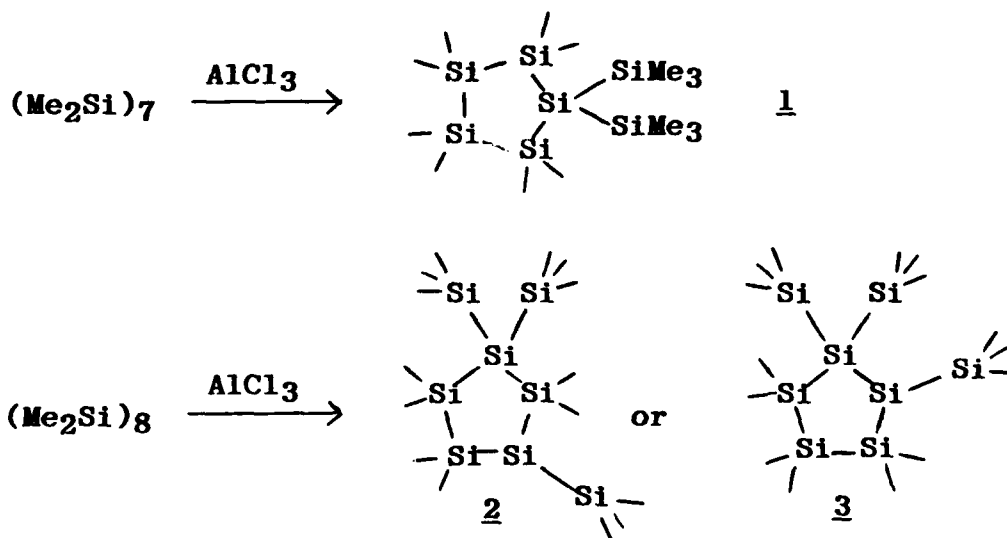
The first light-scattering studies of polysilanes were carried out, partially in collaboration with Drs. Robert Miller and Pat Cottis of IBM Company. The results show that the polysilanes have very high molecular weights, up to 7×10^6 in some samples, and have persistence lengths indicating that they behave as slightly stiffened random coils in solution.

Silicon-29 NMR spectroscopy has been used extensively by our group to study the stereochemistry of polysilane polymers. Polysilane homopolymers with the two organic groups on each silicon identical, such as $(n\text{-Hexyl}_2Si)_n$, show only a single sharp line in the ^{29}Si NMR, indicating that conformational effects on the silicon nucleus are rapidly averaged. Polymers with two different

alkyl groups, such as $(n\text{HexylSiCH}_3)_n$, show symmetrical multiplets of 5-7 closely-spaced lines. These spectra are consistent with a random, atactic arrangement of groups along the polysilane chain. Polymers which contain an aryl and an alkyl group, such as $(\text{PhMeSi})_n$, show three distinct multiplet bands, due to different, nonrandom distributions of stereoisomers along the chain.

In order to elucidate the stereochemical arrangement in $(\text{PhMeSi})_n$, the ^{29}Si NMR of model six-membered ring compounds, $\text{tttttt}-(\text{PhMeSi})_6$ and $\text{ttttcc}-(\text{PhMeSi})_6$, have been carefully investigated. The proton and ^{29}Si spectra for both molecules have been correlated by 2-dimensional NMR studies.

NMR investigations have also been used to determine the structure of some cyclic polysilanes. Cyclic permethylpolysilanes undergo aluminum chloride-catalyzed rearrangement to branched cyclic polysilanes with smaller ring size. For example, $(\text{Me}_2\text{Si})_7$ forms compound 1 under these conditions. The rearrangement of $(\text{Me}_2\text{Si})_8$ leads to a compound whose structure was uncertain between the alternatives 2 and 3:



The structure of rearranged $(\text{Me}_2\text{Si})_8$ was solved by NMR and shown to be 2. This experiment and a similar proof of structure for the rearrangement product from $(\text{Me}_2\text{Si})_9$, represent the first 2-dimensional silicon-silicon NMR spectra ever reported.

A new class of polymers has been synthesized: The disilanylene-ethynylene copolymers, 4a-c. The route of synthesis is shown below:



- 1) $2n\text{BuLi}$
- 2) $\text{ClSiR}'_2\text{SiR}'_2\text{Cl}$



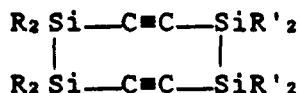
4a, $\text{R} = \text{R}' = \text{CH}_3$

4b, $\text{R} = \text{R}' = n\text{-butyl}$

4c, $\text{R} = \text{CH}_3$, $\text{R}' = n\text{-butyl}$

These polymers are unique in that they show electron delocalization involving mixing of σ and π orbitals. Other electron-delocalized polymers involve only π orbitals (polyacetylenes, polyaniline, etc.) or only σ orbitals (polysilanes). Unusual electronic properties for the new polymers 4a-c are anticipated but remain to be explored.

If ethylmagnesium bromide is used in place of n -butyllithium in the second step of the equation shown above, the products are not polymers but instead are eight-membered rings having structure 5a-c. These novel structures are expected to lead to interesting new types of materials through further reactions.

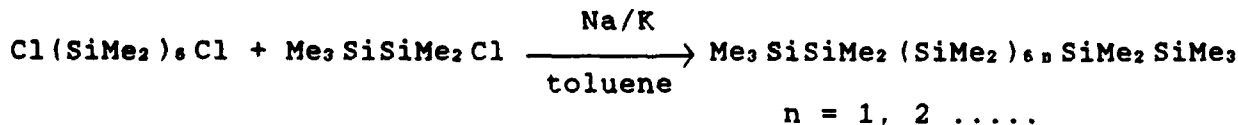
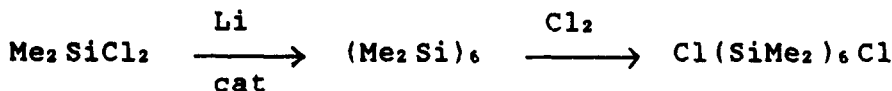


5a, $\text{R} = \text{R}' = \text{CH}_3$

5b, $\text{R} = \text{R}' = n\text{-butyl}$

5c, $\text{R} = \text{CH}_3$, $\text{R}' = n\text{-butyl}$

As models for exploring the electronic properties of polysilanes, some linear oligomeric polysilanes have been synthesized. $\text{Me}(\text{SiMe}_2)_{10}\text{Me}$ and $\text{Me}(\text{SiMe}_2)_{16}\text{Me}$ have been made by the following reactions:



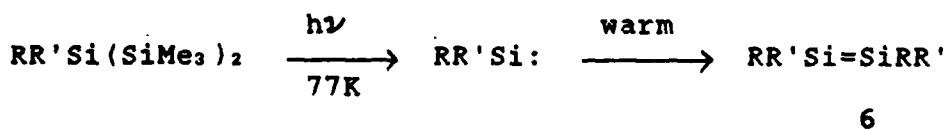
The structure and conformations of these linear oligomers are being studied.

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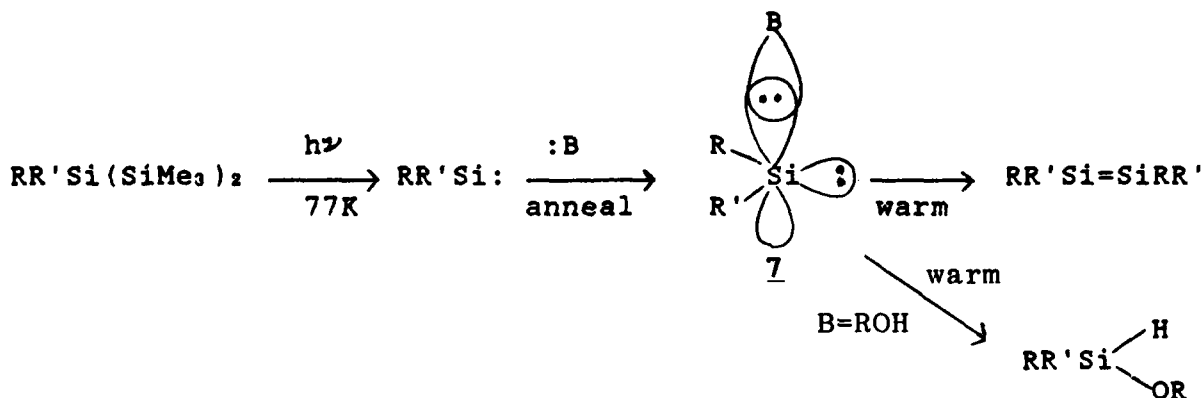
II. Divalent and Multiply-Bonded Silicon Compounds

A. Silylenes, $RR'Si:$

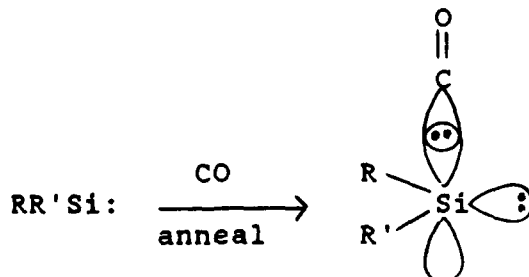
Divalent silicon compounds (silylenes) are highly reactive. Earlier we discovered that silylenes can be generated photochemically in hydrocarbon matrices at liquid nitrogen temperature, and that they are fully stable under these conditions. Many silylenes have now been directly observed by electronic spectroscopy. Warming of the hydrocarbon matrix leads to dimerization of the silylenes to form disilenes, 6.



Recently we have found that if a Lewis base is present with the silylene in the hydrocarbon glass, annealing of the matrix leads first to an acid-base complex 7, which can also be observed by electronic spectroscopy. Bases which form complexes with silylenes include ethers, alcohols, amines, sulfides and phosphines. In most cases further warming of the matrix leads to the disilene, but in the case of alcohols as bases further warming produces a reaction between the disilene and the O-H group of the alcohol.

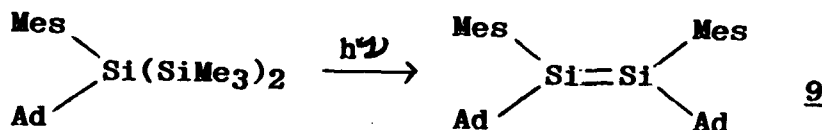
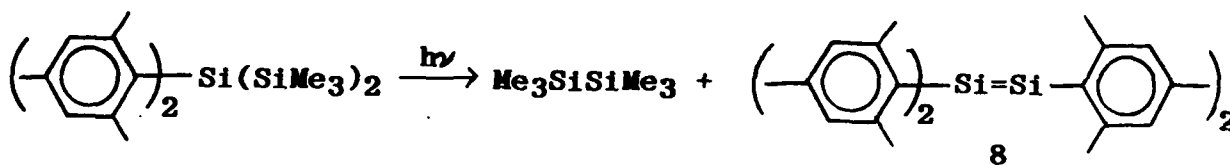


A particularly interesting class of silylene complexes discovered very recently are those between carbon monoxide and silylenes:



B. Disilenes, $R_2Si=SiR_2$

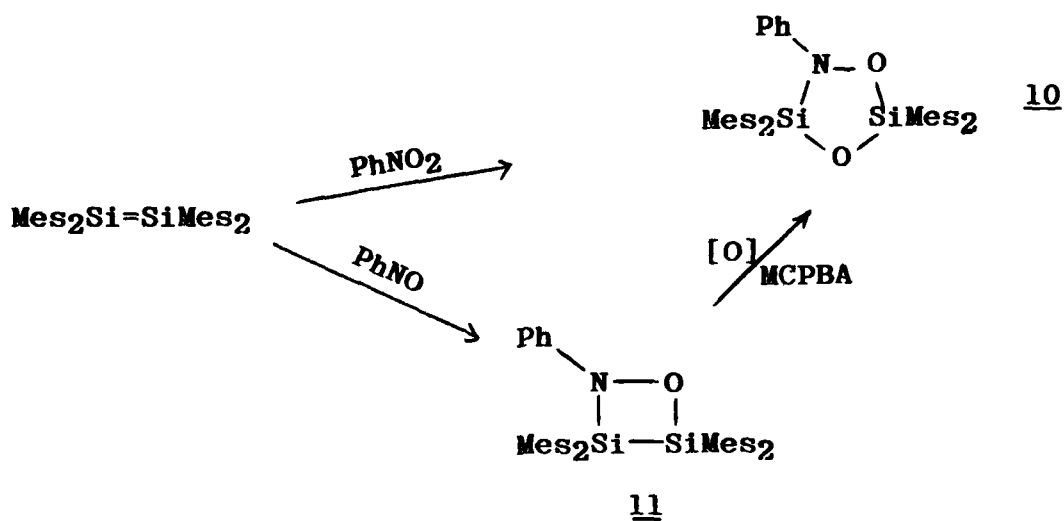
The first compound containing a silicon-silicon double bond, tetramesityldisilene (8), was synthesized at the University of Wisconsin in 1981. Under this contract the chemistry of disilenes has been further explored. The disilenes are synthesized by photolysis of trisilanes. The new, highly stable disilene 9 was obtained by this procedure. Compound 9 survives heating to 275°C and is nearly inert to oxygen.



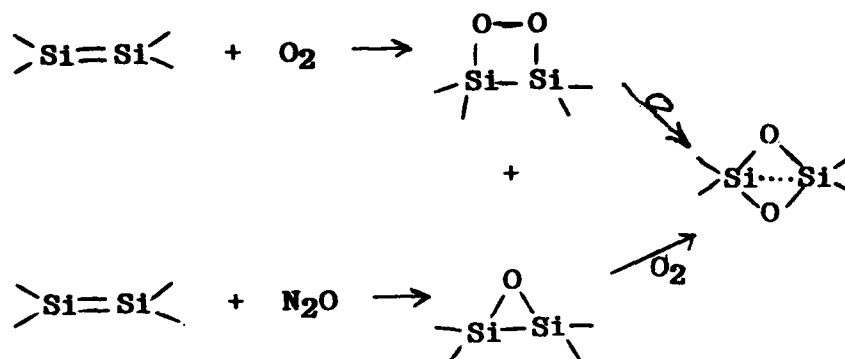
Mes = mesityl; Ad = 1-adamantyl

The activation energy for cis-trans interconversion of 9 was measured by NMR spectroscopy, and found to be 28 kcal mol⁻¹. The crystal structure of 9 has been partially solved by x-ray diffraction.

New reactions of disilenes discovered under the contract include the reaction with nitrobenzene, to give 10; and the reaction with nitrobenzene to give 11, which undergoes further oxidation to 10.



The oxidation of disilenes has been further elucidated. The reaction pathways are illustrated below:



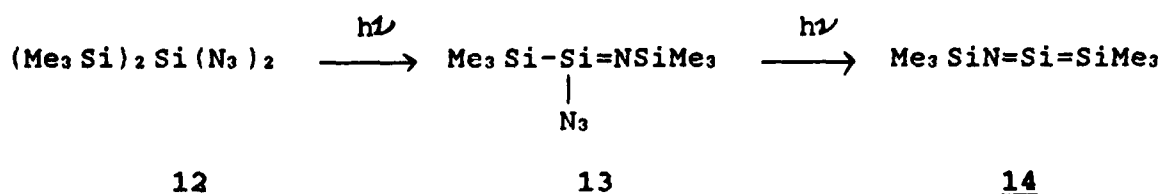
The oxidation products have novel structures with short Si-Si bonds, suggesting possible pi-bonding between the silicons.

The first electrochemical studies of disilenes have been carried out by means of cyclic voltammetry. Oxidation potentials for the disilenes are quite low (+0.5 v) and vary little with substitution at silicon. Reduction potentials are fairly negative, and vary with substituent, indicating that conjugation with aromatic rings influences the energy of the lowest unoccupied molecular orbital in disilenes.

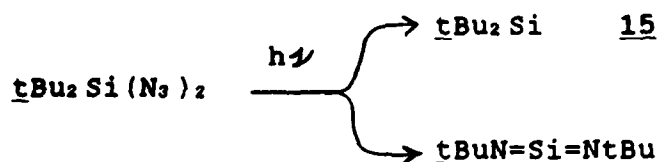
C. Silanediimines, $\text{RN}=\text{Si}=\text{NR}$, and Silanamidines, $\text{RNH}-\text{SiX}=\text{NR}$

Organosilicon compounds in which silicon forms two double bonds to other atoms were completely unknown before the beginning of this contract. Two examples have now been obtained, although both are stable only at low temperatures.

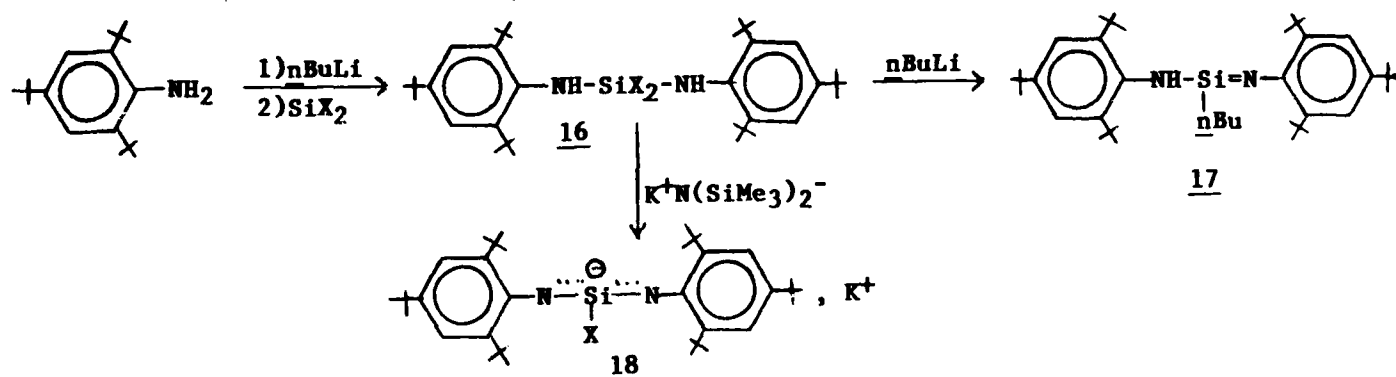
The first silanamide was obtained by photolysis of bis(trimethylsilyl)silyl diazide, 12, in hydrocarbon matrix at 77K. The first product is the silanimine 13, which can be trapped by chemical reaction. Further photolysis converts 13 to the silanediimine 14.



Photolysis of di-tert-butylsilyl diazide also leads to a silanediimine, but in this case the major product is the silylene, 15:



Recently, we have been attempting to make a stable silanediimine. These experiments have led to the synthesis of the first silanamidines (17). The starting compound is the silanediimine 16, made from tri-tert-butylaniline and silicon tetrahalides.



Treatment of 16 with *n*-butyllithium affords silanamidine 17, while deprotonation of 16 with KN(SiMe₃)₂ yields the silanamidine anion, 18. These are the first examples of each of these structural types. Loss of halide from 18 should provide the silandiimine; experiments to bring about this reaction are continuing.

COMPLETED PROJECT SUMMARY

1. **TITLE:** Chemical Reactions and Properties of Organosilicon Compounds Related to New Materials
2. **PRINCIPAL INVESTIGATOR:** Robert West
Department of Chemistry
University of Wisconsin
Madison, WI 53706
3. **INCLUSIVE DATES:** October 1, 1985 - September 30, 1988
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\$241,093, FY87

6. **SENIOR RESEARCH PERSONNEL:**

<u>Name</u>	<u>Present Affiliation</u>
Mary-Ann Pearsall	Drew University, Madison, NJ
Akira Sekiguchi	Tohoku University, Sendai, Japan
Takahisa Iwahara	Kanegafuchi Chem. Industries, Kobe, Japan
Johannes Belzer	University of Wisconsin, Madison, WI
Yuxia Liu	University of Wisconsin, Madison, WI
Li-Ming Huang	University of Wisconsin, Madison, WI
Yoshitaka Hamada	University of Wisconsin, Madison, WI

7. **JUNIOR RESEARCH PERSONNEL:**

Kevin Welsh	Columbia University, New York, NY
Steven S. Zigler	duPont Co., Wilmington, DE
Gregory Jamison	Univ. of North Carolina, Durham, NC
Howard Yokelson	Amoco Co., Naperville, IL
Jim Maxka	Technion, Haifa, Israel*
Gregory Gillette	Univ. of Toulouse, Toulouse, France*
Brian Shepherd	General Electric Co., Schenectady, NY*
Gail Underiner	University of Wisconsin, Madison, WI
Eric Pham	University of Wisconsin, Madison, WI

JUNIOR RESEARCH PERSONNEL (continued)

Alan Fanta	University of Wisconsin, Madison, WI
Tetsuya Asuke	University of Wisconsin, Madison, WI
Tony Millevolte	University of Wisconsin, Madison, WI
Jeff Cavalieri	University of Wisconsin, Madison, WI
Scott Archibald	University of Wisconsin, Madison, WI

*Locations as of January 1, 1989

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44. E-1,2-Dimesityl-1,2-di(tert-butyl)disilene, G. R. Gillette, H. B. Yokelson and R. West, *Organomet. Synth.*, 4, 529-533 (1988).
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9. ABSTRACT OF OBJECTIVES AND ACCOMPLISHMENTS:

I. Polysilane High Polymers and Oligomers. a) Light-scattering studies of molecular weight and chain conformations were carried out for six polysilane samples. b) Silicon-29 NMR spectroscopy was employed to study configurations of polysilane polymers and oligomers. c) A new class of substances, poly(disilanylene-co-ethynylene) copolymers, were synthesized which show properties indicating σ - π electron conjugation.

II. Divalent and Multiply-Bonded Silicon Compounds. a) Complexes of silylenes, $R_2Si:$, with various Lewis bases have been observed spectroscopically. b) Reactions of disilenes ($R_2Si=SiR_2$) with nitro compounds, nitroso compounds, azides and oxygen have been studied, leading to previously unknown types of cyclic silicon compounds. c) A new, very stable disilene, 1,2-dimesityl-1,2-bis(1-adamantyl)disilene, was synthesized and investigated. d) The first silandiimines, $RN=Si=NR$, have been made and observed at low temperatures. e) Stable silanamidines, $RNH-Si(X)=NR$, and silanamidine anions, $RN=Si(X)=NR^-$, have been synthesized and studied.